

A Comparison Between PEG and PVAc as ABS Polymeric Membrane Modifiers for CO₂ Separation from Flue Gases

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Abstract

Low and high molecular weights poly(ethylene glycols) (PEGs), the PEG-400 and PEG-20000, respectively and also, poly(vinyl acetate) (PVAc) with a very high molecular weight of 170000, were considered as the modifiers for acrylonitrile-butadiene-styrene (ABS) terpolymer in CO₂/N₂ separation. The gas permeation and separation properties of the prepared membranes were studied. The results showed that CO₂ permeability as well as CO₂/N₂ selectivity increased for all the blends compared with the pristine ABS. Furthermore, it was revealed that the selectivity enhancements were mostly due to the solubility rather than the diffusivity. Finally, it was concluded that the ABS/PEG blend membranes offered better results than ABS/PVAc ones for capturing CO₂ from flue gases. Among the studied PEGs, the PEG-20000 showed better results than PEG-400. By this modification, CO₂ permeability and CO₂/N₂ selectivity increased from 5.22 and 25.97 for the pristine ABS to 9.76 and 44.36 for the PEG-20000 modified ABS, respectively which caused an increase of nearly 87% in permeability and 71% in selectivity.

Keywords

Polymeric Membrane; Modification; CO₂ Separation; Flue Gas; ABS; PEG; PVAc.

Introduction

The increase in the growing rate of climate change has been worldly accepted. The climate system is obviously shifting towards warming with an increment between 1.4 and 5.8°C by 2100 (Sanaeepur, Sanaeepur et al. 2014). This will be unlikely affecting the world through widespread melting of snow and ice, rising global mean sea levels, loss of biodiversity, transition of ecosystems, and reduction in the value

and variety of agricultural products (Pachauri 2008). Human-made climate change is the main contribution known in this case (VijayaVenkataRaman, Iniyar et al. 2012). The growth of industrial activities in the past century has coincided with a considerable increase in the emissions of so-called "greenhouse gases" (GHGs) in the atmosphere (Ebadi Amooghin, Sanaeepur et al. 2011); and accumulation of these gases in the atmosphere can cause trapping of the heat radiated from the Earth's surface and rising of the Earth's surface temperature, which is known as the "greenhouse effect" (Myers, Luebke et al. 2010; Panwar, Kaushik et al. 2011; Sanaeepur, Ebadi Amooghin et al. 2011).

It is of great global concern to attempt to decrease the amounts of greenhouse gases. This issue has been reflected in two main international documents: the United Nations Framework Convention on Climate Change (UNFCCC) and the Kyoto Protocol. UNFCCC of 1992 has provided a framework for controlling the emission of greenhouse gases (GHGs). In addition, the Kyoto Protocol of 1997 has established a set of quantified limitations on GHG emissions and reduction commitments which have the objective of mitigating emissions of the relevant countries by at least 5% below the 1990 levels to the commitment period during 2008 to 2012 (Ebadi Amooghin, Sanaeepur et al. 2010).

Carbon dioxide (CO₂), methane (CH₄), nitrous oxide (N₂O), hydrofluorocarbons (HFCs), perfluorocarbons (PFCs), and sulfur hexafluoride (SF₆) are the six greenhouse gases addressed in the Kyoto Protocol (Akella, Saini et al. 2009). Among these gases, CO₂

with 77% of the total GHGs emissions has the largest and most determining contribution (FIG. 1) (EPA 2012). Fossil fuels' burning are used in industries for power generation causing the release of huge amounts of flue gases mainly consisting of CO₂. As it is observed in FIG. 1, this portion of CO₂ emission individually comprises beyond half of the GHGs' emissions. This leads to allocation of vast worldwide interests on the carbon dioxide capture and its subsequent storage (CCS) strategies in order to control and reduce its emission (Takht Ravanchi and Kargari 2009; Ebadi amooghin, Zamani Pedram et al. 2013).

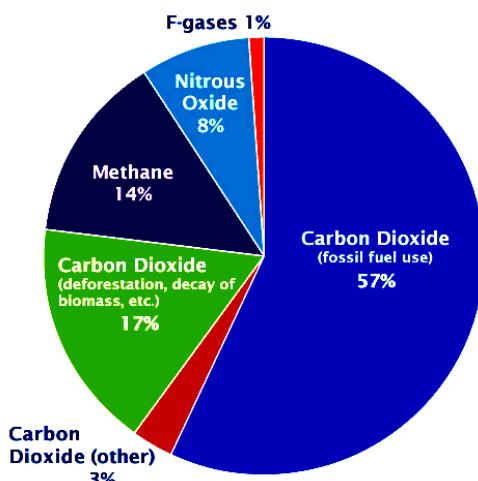


FIG. 1 EMISSION CONTRIBUTIONS OF GLOBAL GREENHOUSE GASES (F-GASES: FLUORINATED GASES WHICH INCLUDE HYDROFLUOROCARBONS (HFCS), PERFLUOROCARBONS (PFCS), AND SULFUR HEXAFLUORIDE (SF₆)) (EPA 2012)

Up to now, different technologies such as physical and chemical absorption, low temperature distillation, pressure swing adsorption and membrane separation have been utilized for CO₂ separation from flue gases (Omidkhah, Zamani Pedram et al. 2013). Membrane separation itself is devoted more favors such as (Kargari and Takht Ravanchi 2012; Arabi Shamsabadi, Kargari et al. 2013; Arabi Shamsabadi, Kargari et al. 2013; Arabi Shamsabadi, Kargari et al. 2014; Mohammadi, Kargari et al. 2014):

1. It neither requires a separating agent nor involves phase changes.
2. No processing costs associated with regeneration and phase change.
3. The systems involve small footprints compared to other processes.
4. They require low maintenance.
5. They are compact and lightweight and can be positioned either horizontally or vertically, which is especially suitable for retrofitting applications.
6. They are modular units and allow for multi-stage

operation.

7. They have linear scale up costs.

Due to its low production costs and reasonable economic performance, (organic) polymeric membrane materials have attracted more interest in comparison with the inorganic materials of membrane gas separation (Sanaeepur, Ebadi amooghin et al. 2010). Most of the existing industrial polymers have been used in membrane preparation. Selecting the material for the membrane fabrication is not arbitrary, but is carried out considering various physical and chemical properties (Sanaeepur, Ebadi amooghin et al. 2011). As permeability is a quantitative criterion of membrane and selectivity is a qualitative criterion, both of them must be appreciated together for membrane design. Up to now, many studies have been carried out to increase the performance of polymeric membranes. Accordingly, the most important methods for increasing the performance of polymeric membranes are as follows (Sanaeepur, Ebadi Amooghin et al. 2012):

1. Incorporation of flexible and polar groups such as amines, carboxyles,
2. Mixing with a carrier (fixed carrier membranes) such as type 1 amino groups such as a CO₂ carrier,
3. Using a soft segment such as poly(dimethyl siloxane),
4. addition of a compatibilizer such as polystyrene-block-poly(methyl methacrylate) in the polymethylmethacrylate/poly methyl ether blend,
5. Polymer blending and interpenetrating polymer networks,
6. Chemical cross-linking and load-bearing network creation via covalent linkages,
7. Structural modification of block copolymers by block copolymerization with a polymer having specific mechanical properties that form a nanostructure, which has physical cross-linkages with favorite properties,
8. Free volume increasing by adding (nano) particles to polymer matrices.

Membrane materials with high CO₂ permeability simultaneous with the high CO₂/N₂ selectivity are of great interest for CO₂ separation from flue gases. The more CO₂ (minor component) selective membrane offers, the less permeation of the major component (N₂) across the membrane, and consequently reduction in overall membrane area required for the separation (Lin, Wagner et al. 2006). Although CO₂ is a nonpolar molecule, it has a higher polarizability and a higher (quadrupole) moment than the other light gases like

N_2 . Therefore, CO_2 solubility increases favorably by the affinity between CO_2 molecules and the polar groups of the polymers (Sanaeepur, Ebadi Amooghin et al. 2012). This increase in solubility is followed by the increase in permeability and so an increase in selectivity. Polymer blending can be a useful, simple, and economical method to satisfy the need for membranes with increased CO_2 permeability/selectivity. Through blending a polymer containing a vast number of weak Lewis basic groups, it is viable to obtain good interactions between them and the weak Lewis acidic CO_2 molecules (Ebadi amooghin, Sanaeepur et al. 2011). Therefore, as the case here, poly(ethylene glycol) (PEG) and poly(vinyl acetate) (PVAc) are considered to blend with acrylonitrile-butadiene-styrene (ABS) terpolymer. PEG with C-O polar bounds in its ether segments can interact favorably with acid gases such as CO_2 , and PVAc with C=O polar bonds in its carbonyl groups can do it as well (FIG. 2).

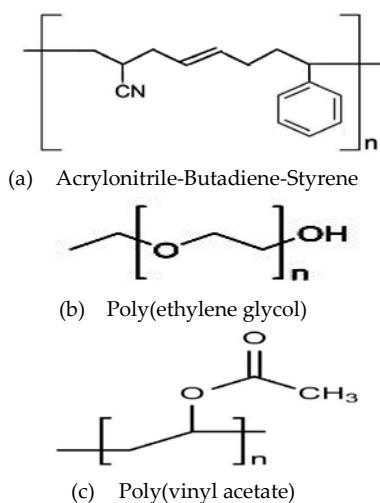


FIG. 2 CHEMICAL STRUCTURES OF (a) ABS (b) PEG AND (c) PVAC

Blending PEG with many polymer membrane materials has been reported by Lin and Freeman (Lin and Freeman 2004). However, in the case of PEG, its strong tendency to crystallize, especially in high molecular weight PEGs, is a great obstruction for gas permeability. A well-known strategy that has been frequently used to overcome the crystallinity increase is the use of low molecular weight liquid PEGs (Lin and Freeman 2005). Therefore a low molecular weight liquid PEG, M.W. 400, is chosen here as the first polyether that will be tested. On the other hand, it has been documented in the work of Li et al. that CO_2 permeability and/or its CO_2/N_2 selectivity of the blend membranes comprising from cellulose acetate and PEGs increase with the increase in PEG molecular

weights up to 20000 (Li, Wang et al. 1998). This is attributed to the increase in the amounts of polyether polar moiety density and its main chain flexibility by an increase in its molecular weight. Accordingly, a high molecular weight solid PEG, M.W. 20000, is the second polyether that is chosen here as the case.

Due to the existence of polar acetate groups in PVAc, its structure varies in such a way that leads to a higher solubility of CO_2 with respect to the other vinyl hydrocarbon polymers (Sanaeepur, Ebadi Amooghin et al. 2011). In the case of PVAc, high molecular weights are desirable to eliminate the molecular weight dependence of the transition temperatures and also to reduce the risk of viscous deformation of the membranes at higher temperatures (Meares 1954). Therefore, a very high molecular weight PVAc, M.W. 170000, is chosen here as the case.

Due to the low mechanical strength of the mentioned polymers, PEG and PVAc, a high enough strength and flexible terpolymer, ABS, was selected as a polymer matrix for fabricating the blend membranes. ABS terpolymer contains acrylonitrile groups that enhance selectivity solubility of CO_2 while decreasing the solubility of nitrogen. On the other hand, due to the presence of double bonds in its butadiene blocks, ABS has good chain flexibility that facilitates the penetrant transport through the polymer matrix. ABS (with rubber domain particle size of $\sim 0.2\mu\text{m}$) will not be an acceptable choice for asymmetric membranes as its domain size is larger than the desired dense film layer ($<0.1\mu\text{m}$). However, its good mechanical characteristic allows preparing a self-existence dense gas separation membrane and/or a dense blend membrane (Sanaeepur, Ebadi Amooghin et al. 2011).

Alongside our previous works on fabricating ABS-based blend membranes with PEGs and PVAc (Ebadi Amooghin, Sanaeepur et al. 2010; Sanaeepur, Ebadi Amooghin et al. 2011; Sanaeepur, Ebadi Amooghin et al. 2012), the present work aims to make a comparison between ABS/PEG and ABS/PVAc blend membranes in the case of CO_2 separation from flue gases. For this purpose, the membranes were compared from the view point of their CO_2/N_2 separation characteristics as well as their diffusivity and solubility, the two molecular transport properties.

Materials And Methods

Materials

ABS terpolymer, containing $\sim 25\%$ acrylonitrile, was

purchased from Aldrich (St. Louis, MO) and was dried about 2h at 80°C in an oven before use. Pure PVAc, with a molecular weight of 170000, was purchased from Acros (Thermo Fisher Scientific, Geel, Belgium). PEG, with a molecular weights of 400 (PEG-400) and 20000 (PEG-20000), were acquired from Merck (Darmstadt, Germany). Dichloromethane (Acros, Thermo Fisher Scientific, Geel, Belgium) was used as solvent without further purification. CO₂, with 99.5% purity, was obtained from Farafan Gas Corp., Tehran, Iran. N₂ with 99.999% purity was supplied by Roham Gas Corp., Tehran, Iran. These gases were used in permeation measurements.

Preparation of the Membrane Samples

The method of solution casting and solvent evaporation was employed to prepare the blend membranes. Polymer solutions with and without PEGs or PVAc were made by dissolving 5wt.% predetermined quantities of the constituent species in dichloromethane as a solvent. To form transparent and homogeneous polymer-casting solutions, we stirred the mixtures for 5h at room temperature and subsequently degassed. The bubble-free solution was cast onto a clean glass plate. After solvent evaporation over a period of 2–3 days at ambient temperature and pressure, the film was removed from the glass plate and further dried in an oven for 12h (in case of liquid state PEG grade used here, drying temperatures for all the blends were considered 30±2°C, and also, the pressures were fixed at 1atm). The resultant defect-free membrane thicknesses were between 18 and 22μm (Ebadi Amooghin, Sanaeepur et al. 2010; Sanaeepur, Ebadi Amooghin et al. 2011; Sanaeepur, Ebadi Amooghin et al. 2012).

Gas Permeation Measurements

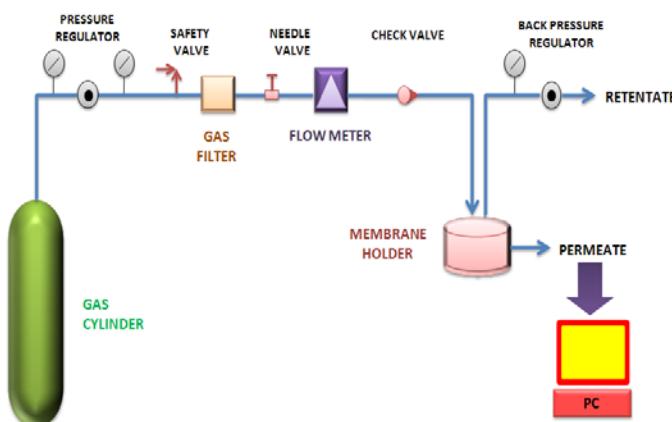


FIG. 3 A SCHEMATIC DIAGRAM OF THE EXPERIMENTAL APPARATUS (EBADI AMOOGHIN, SANAEEPUR ET AL. 2010)

The gas permeability was measured at room temperature at a pressure of 1bar in a permeation test set-up which is shown schematically in FIG. 3 (Ebadi Amooghin, Sanaeepur et al. 2010). A high-pressure stainless steel membrane holder (XX-4504700 from Millipore Co., Billerica, MA) was used with an effective area of 15.90 cm². A glass-stainless steel rotameter (Fisher Co., Hampton, NH) was used for gas flow adjusting. The pressure of the system was kept at the desired value by using a pressure regulator (Parker-Hannifin Co., Mayfield Heights, OH) and a backpressure regulator (GO Co., Spartanburg, SC) both made of stainless steel AISI-316L. Before conducting a measurement, the desired gas stream was established to the cell upstream side, and the transmembrane pressure was kept at the desired value by the backpressure regulator. A 0.003μm Teflon® filter with AISI-316L holder (Gaskleen® 6101 Series from Pall Co., Port Washington, NY) was used in the feed gas line in order to eliminate all of the particles before entering the membrane surface. The permeate flow rate was measured by a precision handmade volumetric flowmeter (Sanaeepur, Ebadi Amooghin et al. 2012). The measured volumetric flow data were used in permeability calculations. In addition, the ideal selectivity related to the measured permeability of pure components was calculated by dividing the permeability of CO₂ over N₂ in the same conditions.

Gas Solubility Measurements

The solubility was measured using a handmade adsorption chamber that consisted of a 100ml high-pressure gas cylinder equipped with a precision pressure test gauge (Bourdon Sedem, Vendome Cedex, France), an electro-polished diaphragm valve (Swagelok Co., Solon, OH), and a high pressure stainless steel membrane holder (XX45 047 00 from Millipore Co.).

X-ray Diffraction

The micro-structural properties of the neat ABS and its blend membranes with 10 wt.% PEGs and PVAc membranes were analyzed on Xpert MPD wide-angle X-ray diffractometer (XRD) from Philips, Holland. The measurements were carried out at room temperature using monochromatic radiation of α-rays emitted by Cu at a wave-length of 1.54Å, accelerating voltage of 40kV, and tube current of 40mA. To identify the crystal structure, the scan range – the angle (2θ) of diffraction– was varied from 1° to 65° with a step increment of 0.025° s⁻¹.

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) thermograms of the ABS and its blend membranes with 10 wt.% PEGs and PVAc were recorded on a PerkinElmer DSC7 instrument. Data were collected from the samples having an average mass of 4mg at a scan rate of 10°C/min from an equilibrated temperature of -110.00°C to a final temperature of 130.00 °C in the nitrogen atmosphere.

Results And Discussion

Permeability and Selectivity

TABLE 1 GAS SEPARATION CHARACTERISTICS OF ABS AND ITS BLENDS WITH 10 WT.% PEG-400, PEG-20000 AND PVAC AT 25 °C AND 1 BAR

Membranes	^a P _{CO₂}	^b α _{CO₂/N₂}	^c D _{CO₂}	^d S _{CO₂}
ABS	5.22	25.97	0.222	235
ABS/PEG-400 (10 wt.%)	5.85	45.70	-	-
ABS/PEG-20000 (10 wt.%)	9.76	44.36	0.250	390
ABS/PVAc (10 wt.%)	5.72	32.5	0.179	320

^a Permeability coefficient; $\times 10^{10}$ [cm³(STP) cm/cm² s cmHg]

^b Ideal selectivity = P_{CO₂}/P_{N₂}

^c Diffusivity coefficient for CO₂; $\times 10^8$ [cm²/s]

^d Solubility coefficient for CO₂; $\times 10^3$ [cm³(STP)/cm³ cmHg]

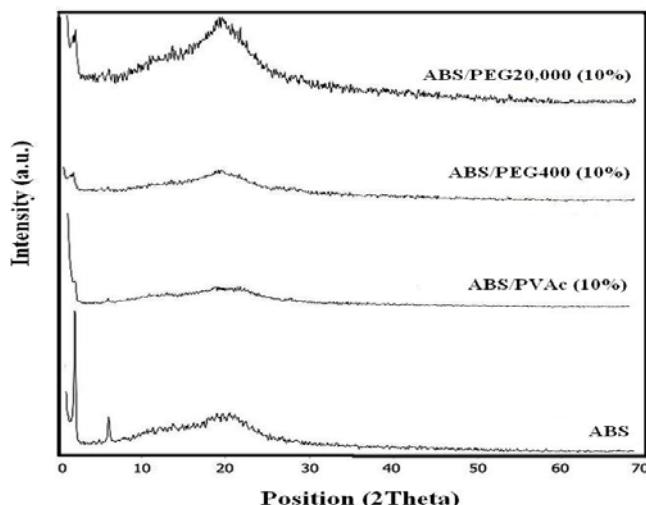


FIG. 4 XRD SPECTRA OF THE MEMBRANES

Gas transport properties of the membrane prepared from ABS and its blends with 10 wt.% PEG-400, PEG-20000 and PVAc (M.W.= 170000) at room temperature and 1bar were tabulated in TABLE 1. All the permeability values are in case of Barrer (1 Barrer = 1×10^{-10} cm³(STP) cm/cm² s cmHg = 7.5×10^{-14} cm³ (STP) cm/cm² s Pa). As it can be seen, PVAc has less effect on both the CO₂ permeability and CO₂/N₂ selectivity compared to PEGs. Therefore, the effects of the two PEGs are further discussed here, and PVAc is considered at the end. CO₂ permeabilities have

increased by the addition of PEGs and by growing in the PEG molecular weights as well. Incorporation of semi-crystalline PEG-20000 in comparison with fairly amorphous PEG-400 has a higher effect on the CO₂ permeability. Although PEG-20000 has more crystalline behavior than the PEG-400, a significant increase in the polyether polar moiety densities by increasing PEG molecular weights from 400 to 20000 boosts the overall interaction of CO₂ molecules with the polymer matrices (FIG. 4), it should be noted that the existence of crystalline zones in the polymer blends raises the peak intensities and also narrows them). This increase in the penetrant-polymer chain interactions can be compensated by the negative effect of polymer crystallinity on the molecular diffusion.

According to TABLE 1, both the blend membranes have higher CO₂/N₂ selectivities than the pristine ABS polymer. In addition, in the case of two blend membranes, CO₂/N₂ selectivity of PEG-400, with a value of 45.7 in relation to 44.36 for PEG-20000, is slightly higher. The addition of PEG-400 to ABS could greatly reduce the overall crystallinity (FIG. 4). A reduction in crystallinity improves CO₂ solubility and the molecular mobility (Patel and Spontak 2004). On the other hand, N₂ is a non-polar permanent gas with linear structured bonds that has no effective polar moment to interact with polymer polar chains. Therefore, N₂ transfer has a diffusion dominant mechanism. As a result, the CO₂/N₂ separation factor of the ABS/PEG-400 membrane has become high.

As it is observed in FIG. 5, with the addition of PEG-400 to ABS, a single glass transition temperature (T_g) – lying in between the two glass transitions of ABS (108.98°C) and PEG-400 (-70°C; that cannot be observed here and adapted from (Feldstein, Roos et al. 2003)) – occurred in the spectrum of the blend. This indicates a superior miscibility between ABS and liquid PEG-400 in the blend, in contrast to the two other spectra of solid PEG-20000 and PVAc. The compositional independence of the glass transitions in the ABS/PVAc blend (two distinctive glass transitions that refer to PVAc at 40.44°C and ABS at 110.32°C respectively) indicates a considerable immiscibility of the two polymers. This is also established for the case of ABS/PEG-20000 wherein a melting peak at ~60°C for PEG-20000 and a T_g peak for ABS is observed.

Comparing the DSC results and the permeability and selectivity data of TABLE 1 demonstrates that the more miscibility in the blends, more suitability for the gas selectivity is obtained. On the other hand,

immiscible blends are more suitable for permeability increase. These findings are in accordance to Bikson et al. (Bikson, Nelson et al. 1994) that expressed that the miscible blends lead to selectivity increase due to the free volume contraction that typically occurs in mixing and results in decreased gas permeation rates. The homogeneous, miscible polymer blends that show a significant negative volume change in mixing will frequently show high gas selectivities but at a significant expense in gas permeation rates.

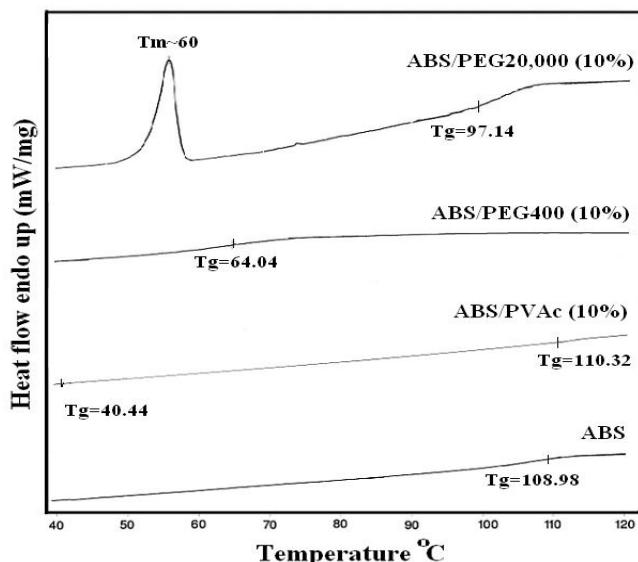


FIG. 5 DSC SPECTRA OF THE MEMBRANES

Diffusivity and Solubility

Incorporation of high molecular weight PEG, i.e., PEG-20000, causes an increase in diffusivity and solubility of CO₂ relative to pristine ABS (TABLE 1). The effect of the PEG molecular weight on the solubility is more considerable rather than the diffusivity. Indeed, the solubility ratio (1.66) for ABS/PEG-20000 relative to neat ABS is more than the corresponding diffusivity ratio (1.13), which indicates the greater effectiveness of solubility. Moreover, this is simultaneously true for the case of high molecular weight PVAc, whereas the solubility ratio (1.36) for ABS/PVAc relative to neat ABS is more than the corresponding diffusivity ratio (0.81), which indicates the greater effectiveness of solubility. However, the diffusivity ratio of 0.81 shows the greater difficulty in the molecular diffusion due to very prolonged chains of PVAc compared to PEG-20000. This can lead to a greater probability of entanglement and consequently trapping the CO₂ molecules and, in turn, declining the diffusivity.

Solubility has a dependency on the chemical structure of the penetrant and membrane and their interactions. C=O polar single bound in ether segments of PEG

have higher electro-negativity than C=O polar double bond in the carbonyl group of PVAc (which is constrained by the electro-negativity reducing effect of the other oxygen in the acetate electron-withdrawing substituent), and thus, it can present stronger interaction with CO₂ acid gas. Therefore, incorporation of PEG-20000 in the ABS polymer matrix causes more enhancements in the CO₂ solubility with respect to PVAc, 1.66 next to 1.36.

Conclusions

PEGs with molecular weights of 400 and 20000 and PVAc with a molecular weight of 170000 were added to ABS to prepare blend membranes for CO₂/N₂ separation. Gas transport characteristics of the membranes were investigated. The results showed that in the case of PEGs, the blend membrane of ABS/PEG-20000 was more suitable for CO₂ separation from N₂ than ABS/PEG-400. Furthermore, an approximately good CO₂/N₂ separation performance with a not-so-good enhancement in CO₂ permeability was observed by the incorporation of PVAc into the ABS matrix. The results are comprised of solubility enhancement rather than the diffusivity's in the presence of C-O polar bounds in ether segments of PEGs as well as the C=O polar bonds in carbonyl groups of PVAc. Finally, it was found that the order of PEG-20000 > PEG-400 > PVAc for the impact of the modifiers on the improvement of permselectivity of the prepared membranes.

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